EXPERIMENTAL STUDY OF SOLIDIFICATION AT EXTREME LOW TEMPERATURE IN A CYLINDRICAL CONTAINER

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EXPERIMENTAL STUDY OF SOLIDIFICATION AT EXTREME LOW TEMPERATURE IN A CYLINDRICAL CONTAINER

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of

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by

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Under the guidance of

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CERTIFICATE

This is to certify that the thesis entitled **Experimental Study of Solidification at Extreme Low Temperature in a Cylindrical Container**, submitted by **Laxman Chauhan** to National Institute of Technology, Rourkela, is an authentic record of debonair research work carried under my supervision and I consider it worthy of consideration for the award of the degree of Master's of Technology of the Institute.

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DECLARATION

I certify that

1. The work contained in the thesis is original and has been done by myself under the general supervision of my supervisor.
2. The work has not been submitted to any other Institute for any degree or diploma.
3. I have followed the guidelines provided by the Institute in writing the thesis.
4. Whenever I have used materials (data, theoretical analysis, and text) from other sources, I have given due credit to them by citing them in the text of the thesis and giving their details in the references.

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At last, I would like to say thank to many known and unknown hands who pushed me forward.

Date :

Place :

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LIST OF SYMBOLS AND ABBREVIATIONS

t_{ice}	Thickness of ice (m)
D	Diameter of cylindrical container (m)
H	Height of the cylindrical container (m)
d_i	Diameter of cylindrical cavity (m)
h	Depth of cylindrical cavity (m)
d_e	Diameter of epoxy compound (m)
LH	Latent heat of solidification or melting (kj/kg)
T_s	Surrounding temperature (K)
T_m	Melting or solidification temperature (K)
c	Specific heat (kj/kgK)
r	Coordinate variable in the radial direction (m)
z	Coordinate variable in the vertical direction (m)
ρ	Density of PCM (kg/m^3)
q	Heat flux (W/m^2)
t	Time (s)
u_r	Velocity component in the radial direction (m/s)
u_z	Velocity component in the vertical direction (m/s)
k_{PCM}	Conductivity of PCM (W/mK)
k_{copper}	Conductivity of cylindrical container (W/mK)
T	Temperature (K)
P	Pressure (N/m^2)

ABSTRACT

The objective of the present paper is to provide the experimental data for the study of solidification of phase change material (PCM) in the cylindrical container. The pure water was considered as a PCM, copper was chosen for the cylindrical container and the liquid nitrogen was taken as the cooling fluid. The experiments were done for different sizes of cylindrical cavities, whose dimensions were 20×20, 20×30, 30×30, 30×40 and 40×40 (diameter in mm$\times$ height in mm). The PCM was taken initially at room temperature for all the cavities. The temperature variation is presented at different time steps during the solidification of PCM. The result shows that the convection from the atmosphere has a significant effect on upper surface. As the diameter of the cavity changes, for the same height, then notable change in solidification time is observed while as the height is changed, for the same internal diameter, the change in solidification time is observed to be less significant.

Keywords: PCM, solidification, conduction and convection, ice track

CHAPTER 1

Introduction

In this thesis, we are dealing with the designing of experimental setup, temperature measurement and developing the idea for ice thickness measurement during the solidification of Phase Change Material (PCM). Many researcher have studied the solidification of Phase Change Material (PCM) but very few of them have reported the study on solidification at extreme low temperature.

Solidification is the process in which the liquid converts to solid and release some amount of energy, that energy is called the latent energy of solidification [2]. This happens when the temperature of PCM is lower than its freezing point. Generally solidification happens at constant temperature for pure materials, but under certain conditions some materials, e.g impure water, solidify within a temperature range. Water being a pure material solidifies at 0^oC and releases 334 kJ/kg energy.

PCM can be in three states, i.e. solid, liquid and gas. It changes from one state to another state, i.e. solid to liquid, liquid to gas or gas to solid or vise-versa. Depending on this, a process may be endothermic or exothermic as shown in figure 1.1. Sensible heat and latent heat are the important terms which are discussed in scientific articles related to solidification. Sensible heat occurs within the range of temperature not at a

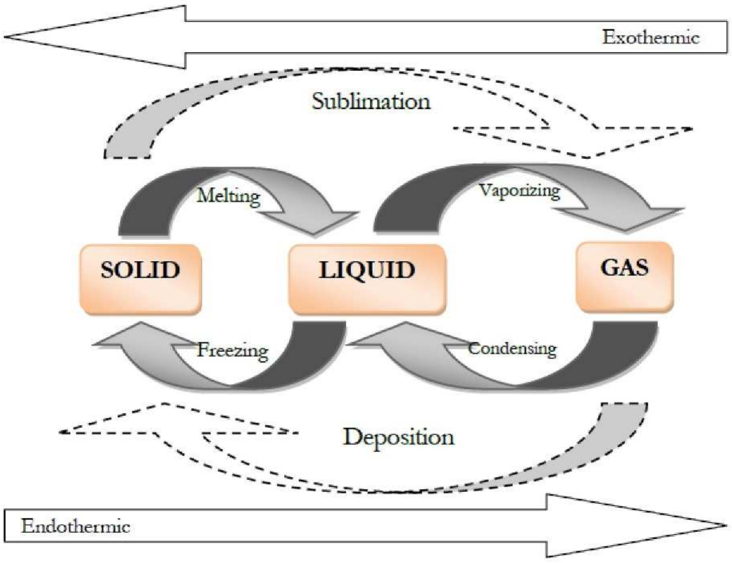


Figure 1.1: PCM transformation phases, adopted from O.A. obitayo's thesis [1]

particular temperature.

1.1 Phase Change Material (PCM)

A number of materials are available and each have its own specific property and area of application [3]. The PCM is classified into 3 main categories i.e. organic, inorganic and eutectic PCM. The detailed classification is given in the figure 1.2. Each material has its

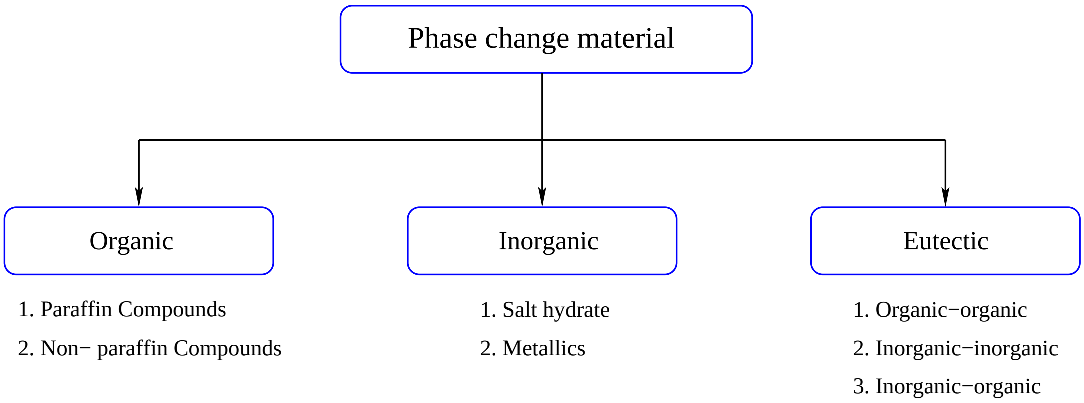


Figure 1.2: Classification of Phase Change Material (PCM)

specific phase change temperature or a range of temperature at which it solidifies. The organic and inorganic PCMs are mainly used because of their properties as shown in table 1.1. Most of the material solidifies in some temperature range, this range is called

Table 1.1: Properties of the organic and inorganic compound

	Merits	Demerits
Organic Compounds	Exist in wide temperature range	Low thermal conductivity in solid state
	Freeze without much super cooling	Flammable
	Compatible with conventional building materials	Low volumetric latent heat storage capacity
	Chemically and thermally stable	Low phase change enthalpy
	High Latent heat of fusion Recyclable Non-reactive and safe	
Inorganic Compounds	Low cost and readily available	Super-cooling
	High Thermal conductivity	High volume change
	Non-flammable	Segregation
	High volumetric latent heat storage capacity	Reduce in efficiency after repeated use

mushy zone. This type of material has different melting and solidification temperature, but for the pure material there is no difference between solidification temperature and melting temperature. Mostly, wax is used as a PCM by researcher's because it is easy to study and it solidifies at room temperature, so there is no need to maintain uniform temperature in a system.

1.1.1 Application

The heat transfer characteristics of a phase change material have been studied from many year because of its broad application in the engineering fields. The advantage of heat storage and heat release capacity of PCM has been studied by many investigators.

Gu et al. [4] used paraffin based PCM for heat recovery from air conditioning (AC) systems and for generating low temperature hot water. If the condenser of an AC is made to be in contact with the PCM then the PCM will melt by taking heat from the condenser and transfer it to water. Thus, PCM act as a medium which takes heat from condenser and transfer it to water. Although, we can heat water directly without using

PCM, but the water will not be at constant temperature and a requirement of big tank also forces us to go for the use of PCM.

The concept of latent heat is used for cooling of portable hand held electronic devices like laptop, mobile, tablet etc. proposed by Fok et al. [5]. Now a days, multimedia mobile has cameras, multimedia facility, web browsing facility, GPS, etc. which generate excessive heat. If the rear side of the mobile is covered with the PCM arrangement, then it will take heat from mobile and transfer to the atmosphere. Thus a constant temperature will be maintained during usage.

Tay et al. [6] used PCM for cooling and heating the building during day and night time respectively. During day time, solid PCM will melt slowly and water is circulated over the PCM and consequently water gets cooled at the exit. During night time, liquid PCM becomes solid and the exit water from the PCM becomes cold. This cold and hot water can be used for the room cooling and heating.

Husseina et al. [7] used PCM in solar cooking system. In such solar cooker a special PCM is used, that is Stearic acid, whose melting point is 70^oC. This PCM based solar cooker can be used even in evenings. During day time whole PCM will melt and in evening time it slowly solidifies. When this slow solidification takes place, latent heat is released which can be used for cooking foods.

Some other example include the production of modern textile products by PCM [8]. Liu et al. [9] first time incorporated PCM into the refrigeration system for refrigerated trucks and casting of steel slabs is done by Manojlovic [10].

1.1.2 Water as a PCM

Selection of the PCM is mostly dependent on the physical, economical, thermal and chemical characteristics. Mostly, in literature, we have noticed paraffin wax is used for the study of solidification but in our experiment water is chosen for the study of solidification at extremely low temperature. Water is an inorganic substance. Generally, when the material solidify its volume reduces, but for water opposite behaviour is observed. When water turns to ice its volume increases. Water is a transparent fluid and it is formed when two hydrogen and one oxygen atom are bonded by covalent bonds. This molecule of water are very close to each other i.e the molecules are in a closed pack. This molecules are bonded by hydrogen atom. When water cools, its density increases and at 4^oC, density becomes maximum. But further cooling results in decrease in density. At 0^oC, water becomes solid and volume increases because the water molecules are arranged in a chain form, like a cube or a tetrahedron structure [11]. The properties of water are given in the table 1.2.

Table 1.2: Property of water

Property	values
Melting point (^oC)	0
Boiling point (^oC)	100
Latent heat of fusion (kj/kg)	333.6
Specific heat at constant pressure (kJ/kgK)	2.05
Specific heat at constant pressure solid (kJ/kgK)	4.186
Maximum density liquid (kg/m^3)	1000
Density at solid (kg/m^3)	917
Thermal conductivity (W/mK)	1.6 - 2.22

1.2 Liquid nitrogen

Liquid nitrogen is an cryogen whose boiling point is around -196^oC. It is colorless, nonflammable and inert gas. Nitrogen is freely and abundantly available. So the best source for producing liquid nitrogen is the atmosphere. Liquid nitrogen is prepared from the Linde cryo-plant by separating the nitrogen from the air and then by condensing the nitrogen gas.

1.3 Review of Literature

Various numerical techniques have been used in the past for investigation of solidification in different geometries. Smith et al. [12] studied the solidification of PCM numerically inside a thick wall cylindrical container using an alternating-direction-implicit method for solving the governing equation. A similar problem with some extra varying parameters have been done by Tien and Chang [13].

Caldwell and Chan [14] applied the enthalpy method for the numerical study of solidification in a spherical geometry and compared their results with the heat balance integral method (HBIM) for a wide range of Stefan numbers. Smail and Jesus [15] dealt with solidification of PCM around a cylinder, carrying a heat transfer fluid inside. They considered a pure conduction model for the entire system. Dubovsky et al. [16] also studied the process of solidification of a phase change material (PCM) in cylindrical shells using Fluent 6.2.

Tan and Leong [17] have done an experimental study of the conjugate solidification process of n-octadecane as the phase change material inside a thick cylindrical mold considering constant base temperature for different superheated PCMs. They have considered two different materials, i.e. brass and stainless steel, for their studies and found that the solidification process is faster in brass as compared to stainless steel. They concluded that the solidification mass fraction is directly proportional to the cube root of solidification time for sub-cooled wall condition.

Lipnicki [18] has done an experimental analysis of solidification of water with blue methylene in an annular enclosure. He compared the experimental result with the analytically result and gave a good correlation between them. Generally CFD helps to trace the solidification front, but Lipniki used a transparent cylindrical medium and a binary solution of water with methylene blue for visualization of solidification front.

Smith and Meeks [19] have done experimental and numerical studies to provide quantitative data for a simple multidimensional solidification process of n-octadecan (PCM) in an enclosure. They presented the shape of the phase front profile. Elgafy et al. [20] have studied the solidification and melting of high melting point phase change material (aluminum) numerically and experimental by considering convection as well as the radiation effects.

Jones et al. [21] have done experimental measurements during the melting of a moderate-Prandtl number material (paraffin wax, n-eicosane) in a cylindrical enclosure and gave the benchmark experimental measurements for validation of numerical codes. As for the numerical solution, finite volume method is used and a second order implicit scheme was used for the transient term while the second order upwind scheme was used for the convective term and central differentiating was used for the diffusive / conductive term. The multi-block method was used for solid and liquid regions.

Sharifi et al. [22] studied the effect of the inclination of a cylindrical enclosure on the heat transfer characteristics during melting of PCM (a paraffin wax, n-octadecane). Kamkari et al. [23] have done experimental analysis of melting of phase change material similar to Sharifi et al. [22] but studied at particular three angles, i.e. 0^o,$45^o$and 90^o, and found that the melting time required for 45^o and 0^o was 35% and 53% less than the time required for 90^o enclosure.

Some of the authors investigated melting of PCM experimentally and numerically. Shrivastava et al. [24] have done numerical investigation of melting using computational fluid dynamics (CFD) in a vertical cylindrical geometry considering the internal heat generation. They also obtained the experimental result of melting. Shmueli et al. [25] compared the numerical solution to the previous experimental solution of melting of a PCM in a vertical circular tube.

Although many researchers have studied the solidification or melting of PCM in different geometries such as cylinder, annular tube, spherical shell, etc. But none of the studies reported solidification of a PCM in a cylindrical container whose surface is maintained at an extremely lower temperature. This paper investigates solidification of a PCM in a cylindrical container by a low boiling point liquid as a heat transfer fluid. The intention of the present work is to investigate and predict experimentally the thermal behavior of phase change material during the solidification process within a cylindrical container at extreme lower temperature boundary condition, i.e. low boiling point liquid (liquid nitrogen).

1.4 Outline of the thesis

Developing an experimental setup and performing various experiments for the parametric studies for the solidification are the main focus of the present study. The main work of this thesis is to explore the heat transfer characterstics of solidification for all cylinder containers and trace the ice thickness in each cylindrical cavity. This thesis consists of five chapters and the brief description of each chapter is given below.

Chapter 1: This chapter contains the general background of the historical necessity of study the solidification. It is started with a few some simple terms like solidification, latent heat, sensible heat and the classification of PCM and its application in various fields, concept of latent heat, literature review and the objective of the study.

Chapter 2: This chapter deals with the theoretical background and some important governing equations. This chapter also informs about various methods for solving the solidification problem. The last paragraph in this chapter explains the current solidification problem and its boundary condition.

Chapter 3: This chapter fully explains that how to make experimental setup and how to do experiments. It explains the history of the development of the setup which satisfy all boundary conditions. The main part of the setup is explained separately. It also explains the procedure of the temperature measurement and the methods adopted for ice tracking.

Chapter 4: In this chapter, the results obtained from the study are discussed and the ice thickness is shown for each cavity at 40% and 65% of complete solidification. It also presents the relationship between depth and internal diameter of the cavity and on the basis of the result, the conclusions are drawn.

Chapter 5: Summary of the whole thesis is in this chapter. The direction of future work has been presented.

CHAPTER 2

Theoretical Background

The solidification or melting is related to Stefan problem. Stefan has done the study of melting of the polar ice. The phase change problem is also called moving boundary problem (MBP) as the interface of ice and water moves with time as solidification or melting takes place [26][27]. The moving boundary problems are nonlinear in nature and more complex. Hence, the exact solution of such problems are difficult to obtain for 2 or 3 dimensional problems. But for 1-D problems, Crank [27] obtained results which are more accurate. In actual practice, most of the solidification and melting problems are 2 dimensional or 3 dimensional, only few of them can be simplified enough to be one dimensional. Boundary conditions are also complex and physical properties change with respect to time. The other ways to solve this problem are numerical and experimental solutions.

2.1 Analytical solution

The analytical method can be used for some simplified problem of solidification. If the problem is one dimensional and the boundary conditions are simple then analytical methods can be used. For example, Stefan[26] solved a solidification problem for one phase and derived a non dimensional number.

$$\text{Stefan no.} = \frac{[C(T_S - T_m)]}{LH}$$

Where LH = Latent heat

It gives the relationship between sensible heat and latent heat of PCM.

Stefan gave the analytical solution only for the one phase (1989-91) [28] related to melting of polar ice cap. After that, many studies were reported on the analytical study of solidification and melting, but all of those problems were considered to have simple boundary conditions.

2.2 Numerical solution

As an analytical method has some limitations, researchers are giving more preference to the alternate solution, i.e. Numerical solution. Numerical methods are used to solve complex problems. There are various numerical methods to solve moving boundary problem. The numerical method can be classified into three group, i.e. fixed grid methods, variable grid methods and method of latent heat evolution. The detailed classification of numerical methods for solidification and melting problem is shown in the figure 2.1. Finite volume method, finite element method and finite difference

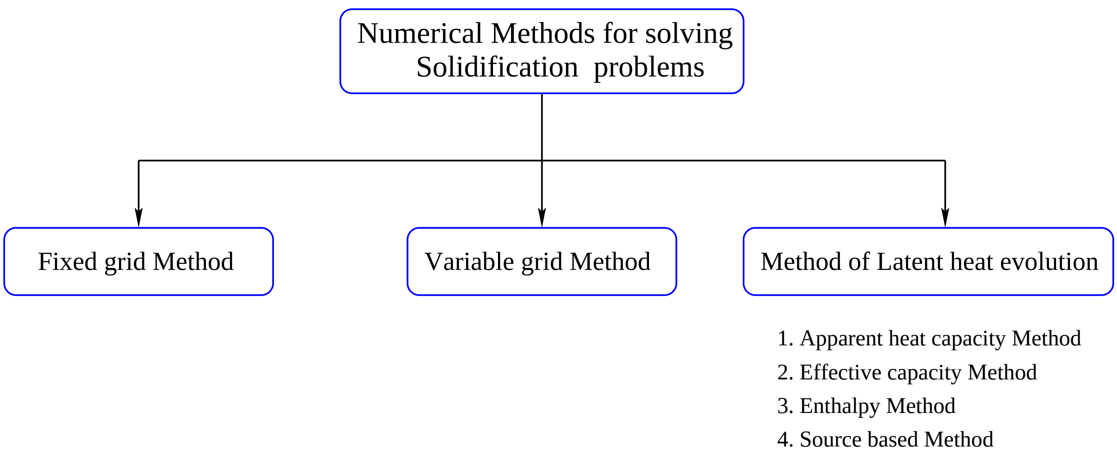


Figure 2.1: Various numerical method for solving solidification and melting problems

methods are the various methods used for the discritization of the governing equation.

Table 2.1: Boundary condition

Boundary	Boundary condition	Range
$r = 0$	$\frac{\partial T}{\partial r} = 0$	for $z = 0$ to $z = H$
$r = R$	$T = T_{LN}$	for $z = 0$ to $z = H/2$ from the base
$r = R$	$T = T_{VN}$	for $z = 0$ to $z = H/2$ from the top
$z = 0$	$Q = 0$	for $r = 0$ to $r = R$
$z = H$	$h_a(T - T_a) = k\frac{\partial T}{\partial Z}$	for $r = 0$ to $r = R$

2.3 Experimental method

Experimental study is an another method for solving the solidification problem. Experimental study is a way of study in which all actual conditions are considered. This thesis mainly deals with the experimental study of solidification in a copper container. It considers actual boundary condition and the detail explanation of the problem is given in the next paragraph.

2.3.1 Current problem explanation

Every problem, it may be either experimental or numerical, has some constrains. If numerical approach is adopted, the boundary condition is easy to maintain uniform throughout the study, but for the experimental study, maintaining the uniform boundary is tedious and complex task. In experimental studies initial focus is towards making the experimental setup ready because the setup should be robust and precise results are expected from the experiment.

For all the experiments, the base of the copper container was kept in the cylindrical styrofoam container so as to minimize the heat transfer from the base. The bottom half of the container was kept in contact with the liquid nitrogen, i.e. up to 30 mm from the base was at -196 °C. The top of the cylindrical container was in direct contact with the atmosphere and the atmospheric temperature was taken as 22 to 23 °C for each experiment. The boundary condition of the copper blocks are represented in a schematic 2D domain and in the tabular form as shown in figure 2.2 and table 2.1 respectively.

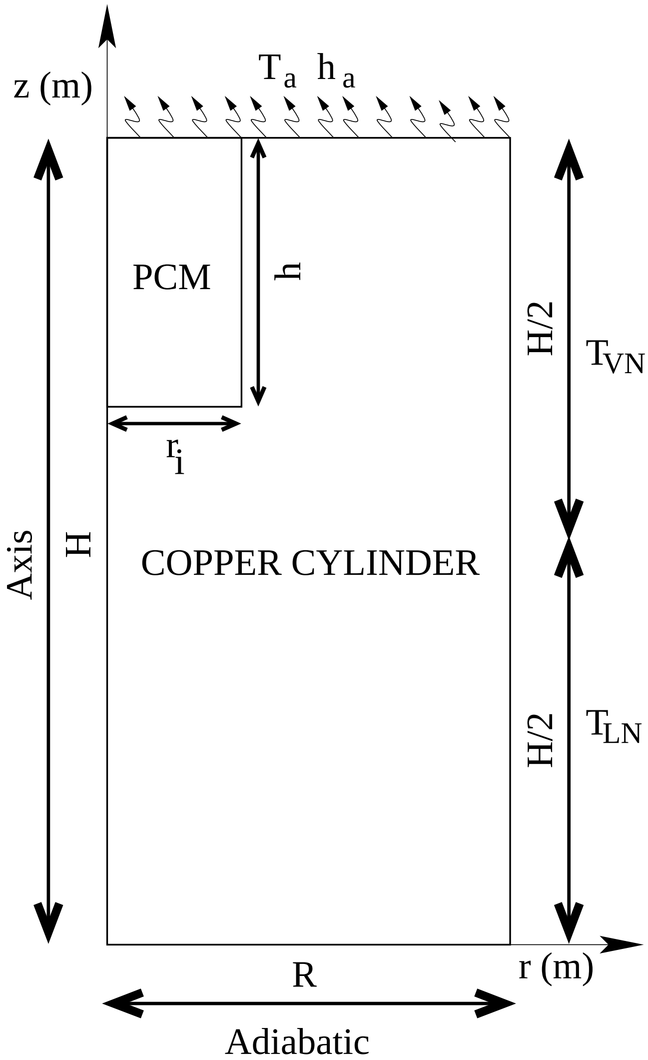


Figure 2.2: Schematic drawing of a 2D copper domain with boundary condition

CHAPTER 3

Experimental set-up and procedures

3.1 Experiment setup

As for maintaining all boundary condition the setup was prepared. It satisfies all conditions, according to the our requirement. The study of solidification is done in several copper containers, therefore five copper blocks are machined. The setup consists of four main part, i.e. distribution tank, double cylindrical Styrofoam container, tripod stand, thermocouples with DAQ arrangement.

3.1.1 Copper containers

The cylindrical copper rod of 60 mm diameter was purchased and after some machining operations, i.e. cutting, drilling, milling and turning respectively, five copper containers were made each having an external diameter and height of 60 mm. Each copper container has a symmetrical cylindrical cavity of different dimensions which was used for the experiments as shown in figure 3.1. To study the effect of cavity height and diameter, they are varied as 20×20, 20×30, 30×30, 30×40 and 40×40. The cavity dimensions are expressed as diameter in $mm \times$height in mm.

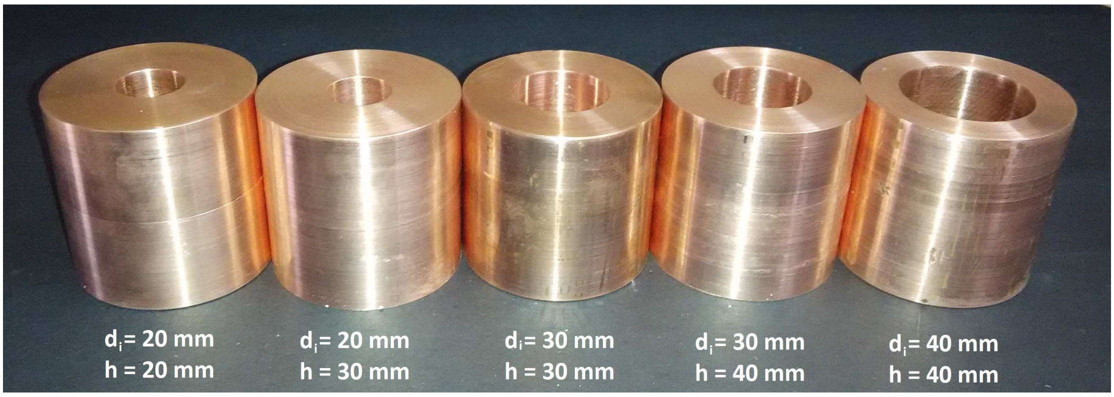
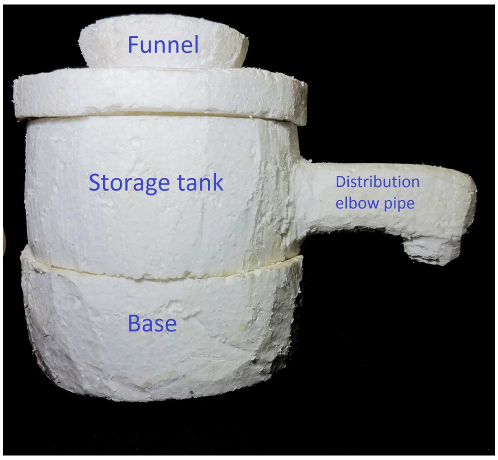
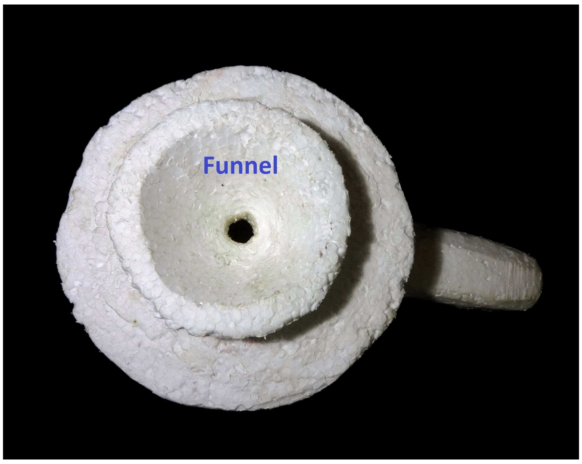


Figure 3.1: Copper containers



(a) Front view of distribution tank



(b) Top view of distribution tank

Figure 3.2: Distribution tank

3.1.2 Distribution tank

The distribution tank made from the 70mm thick styrofoam slab. The function of the distribution tank is to provide continuous and uniform flow of liquid nitrogen to main cylindrical styrofoam container. The other simple way is to provide the liquid nitrogen through a small diameter pipe, but in this case the outlet pressure and velocity will not be uniform and also some particle of liquid nitrogen spitting on the upper surface of copper blocks. So for avoiding this problem and maintaining a boundary condition distribution tank was prepared. It consists of three parts base, small capacity storage tank with distribution elbow pipe and a funnel as shown in figure 3.2. The small storage tank is open only at inlet and outlet. The storage tank is mounted on the base and the head of the storage tank, connect to funnel while the other end is the extended part in the form of an elbow pipe. The internal diameter of the elbow pipe is 5 mm. The internal height of the storage tank in $50mm$ and the distribution pipe is extended from above the $10mm$ of the base of the storage tank so that outlet liquid nitrogen as minimum as possible of turbulent nature and also uniform flow rate throughout the experiment. The function of the funnel is to provide liquid nitrogen to the storage tank without any wastage. The liquid nitrogen was chosen as a heat transfer fluid and maintain extremely low and constant temperature at the periphery of the copper container.

3.1.3 Double cylindrical Styrofoam container

The double cylindrical styrofoam container is made from 70 mm thick styrofoam slab. The main objective of making the double cylindrical Styrofoam container is to maintain the level of the liquid nitrogen to a particular height i.e. $30mm$ from the base. Although single cylindrical container is enough for liquid nitrogen to be in contact with the copper blocks, this will cause the level of liquid nitrogen in the container which will not be constant. It level will vary continuously about the the mean height ($30mm$ from the base) during the experiment. A passage was given at the height of $30mm$ from the base of the copper block between two cylindrical container so that extra liquid nitrogen goes out from the main cylindrical container and delivers it to collecting container, as the level of liquid nitrogen is more than the mean height. The thickness of the double cylindrical container was made $15mm$. The double cylindrical styrofoam container shown in figure 3.3.

Styrofoam

There are many materials available with low thermal conductivity but Styrofoam is cheaper and easily available material. It is also called expandable Polystyrene. It was invented by Ray McIntire during World War II. As the boiling point of the liquid nitrogen is around-196^oC and the atmospheric temperature is around22^oC to 23^oC. The temperature difference between atmosphere and liquid nitrogen is very high and this causes high vaporization. It is difficult to maintain the level of liquid nitrogen in the

Figure 3.3: Double cylindrical Styrofoam container

double cylindrical Styrofoam container. It also reduces the vaporization of liquid nitrogen.

Some characterstics of Styrofoam are –

1. Lightweight
2. Good formability
3. Good insulator (thermal conductivity = 0.032 to 0.038W/mK)
4. Enough rigidity and shock absorber
5. Easily available

3.1.4 Thermocouples and DAQ arrangements

The function of the thermocouple is to measure temperature. The hardware-software arrangement is used for online monitoring of temperature variation. Seven k-type thermocouples were used for measuring the temperature of copper container and solidification front by placing them at several locations. The five k-type thermocouples were used for measuring the temperature variation across the height of the copper container; these were placed at the height of 10 mm, 20 mm, 30 mm, 40 mm and 50 mm from the base. The thermocouples are fixed by using copper wire as shown in figure 3.4.

Figure 3.4: Arrangement of themocouples in the copper container

. Where 1, 2, 3, 4 and 5 represent five thermocouples at $10mm$, $20mm$, $30mm$, $40mm$ and $50mm$ from the top, respectively. Two thermocouples were used for measuring the temperature variation of PCM with respect to time and placed co-axially at the top and half of the internal height. To provide the rigidity to the thermocouples, these were inserted into the plastic pipe. All seven thermocouples were connected to a DAQ i.e. 16 Channel NI-9213 Data acquisition System and LabVIEW software is utilized for online monitoring of the temperature variation.

3.1.5 Tripod stand

A wooden tripod stand with a center hole is prepared for giving the stability to the plastic pipe. Now the plastic pipe with thermocouple wire was inserted and tight fit into the center-hole of tripod stand. Two thermocouples were inserted into the plastic pipe for place the thermocouple axially and providing the rigidity inside the cylindrical cavity. The plastic pipe with thermocouples wire was inserted and tightly fitted into the center-hole of tripod stand and then the thermocouple wire is connected to DAQ hardware and the DAQ connect to a computer. The arrangement of thermocouple wire with tripod stand is as shown in figure 3.5.

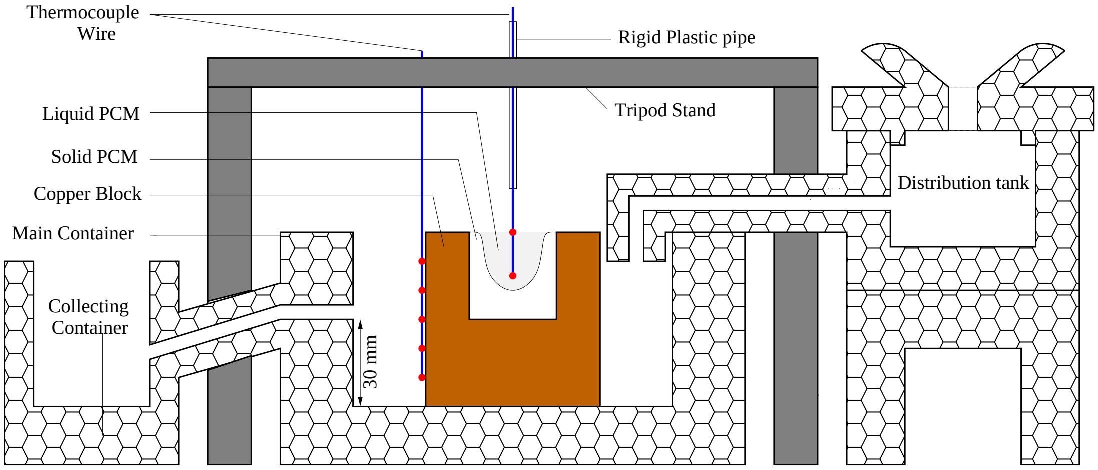
Figure 3.5: Tripod stand

3.2 Procedures

As the aim is to online monitoring and record the temperature history during the whole experiment and ice thickness at different time step that is why there is little change between procedures for the temperature measurement and procedure for the ice thickness measurement. Both the procedures are written ahead.

3.2.1 Procedures for temperature measurement

The copper container filled with distilled water is kept in a styrofoam container. The distribution tank, tripod stand with thermocouples, DAQ and container are kept in a systematic manner as shown in figure 3.6. The thermocouples were connected to PC based Data acquisition system. The distribution tank is kept filled with liquid nitrogen continuously throughout the experiment. Liquid nitrogen comes into styrofoam main cylindrical container through the connecting pipe. The main container is always filled



(a) Schematic setup

(b) Experimental setup

Figure 3.6: Setup

with liquid nitrogen up to the height of 30mm from the base and the extra liquid nitrogen is allowed to flow to the collecting container. The level of the liquid nitrogen in the main container is kept fixed till the complete solidification of the PCM. The temperature was recorded by the use of thermocouple and DAQ arrangement during the experiment.

3.2.2 Procedures for ice thickness measurement

The setup arrangement was again same as for temperature measurement, but this time, the tripod stand and the thermocouples arrangement were not used. Liquid nitrogen is filled in the distribution tank similar to former one, but the experiment was done in the intermittent manner. The solidification time was noted from the temperature reading and the experiment is carried out up to 40% and 65% of the solidification time. As soon as the solidification time reaches 40% of the total solidification time, the copper block is taken out and the remaining water is removed instantly by simply inverting the copper block. Now, the cavity created by the frozen part is filled with epoxy compound. Because of its malleable property, the epoxy compound takes the shape of the cavity. At this time, it was not possible to take out the epoxy compound from the frozen part. The epoxy compound is only taken out when the frozen part remelts completely. Similar procedure is followed for the 65% of the total solidification time experiment. The epoxy compound statue for all the cases are shown in figure 3.7.

The diameter of epoxy compound is measured at different heights, usually at an interval of 2 mm from the top by using vernier caliper.

The thickness of ice at different heights was calculated by using the simple formula –

$$t_{ice} = \frac{(d_i - d_e)}{2}$$

The procedure either for temperature measurement or for ice thickness measurement are repeated several times for each cylindrical container so that more accurate results can be obtained.

(a) Epoxy compound statues at 40% solidification of total solidification time

(b) Epoxy compound statues at 65% solidification of total solidification time

Figure 3.7: Epoxy compound statues

CHAPTER 4

Results and Discussion

As mentioned above, this paper reports an experimental study of the solidification of water inside the copper containers. This section presents some results like temperature history during solidification process, solidified zone at different time instants and the phase front profile at different times for all the studied cases. Altogether five copper blocks have been taken with varying internal cavity dimensions as 20×20, 20×30, 30×30, 30×40 and 40×40.

4.1 Temperature variation

4.1.1 Solidification temperature according to mid thermocouple

The temperature vs time graphs for the axially mid thermocouple reading is shown in the figure 4.1. The cooling medium was liquid nitrogen whose boiling point is $-196^{o}C$ while initially the copper container and water were at room temperature ($22^{o}C$). Because of the high heat content of liquid nitrogen, as soon as it is poured into the main container, the copper block attains the temperature of liquid nitrogen within few sec-

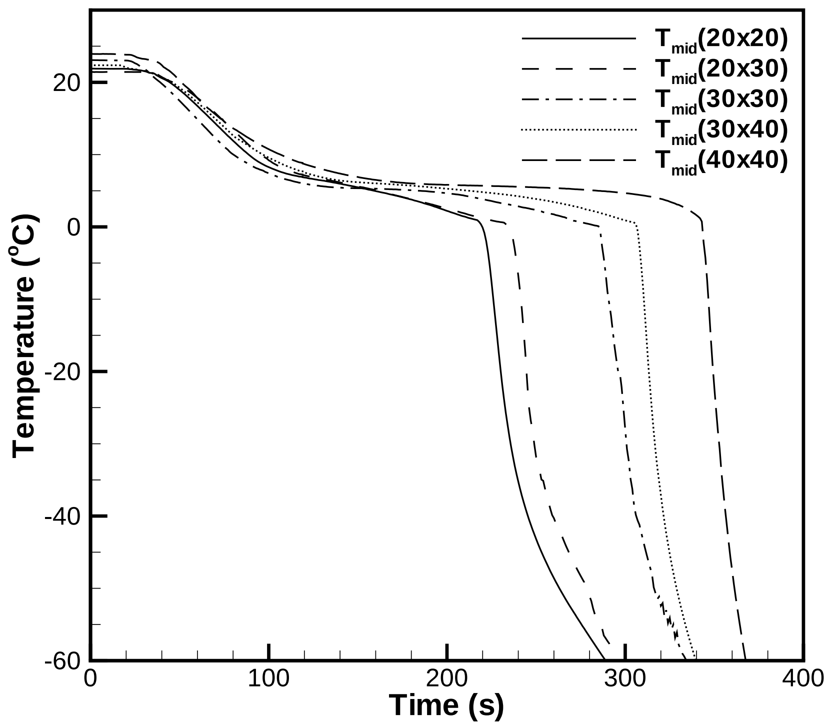


Figure 4.1: Temperature history for the solidification of PCM in different containers for mid thermocouple

onds. This heat transfer causes a high temperature gradient and that is why the temperature drop is fast in the initial stage of cooling.

From the graph, the temperature profile of the PCM for all the containers decreases continuously and follows approximately same pattern up to 100 s. The temperature profile for the 20×20 and 20×30 follows the same pattern, but the 20×30 copper container has taken a little more time. It is noticed that the solidification time increases with increase in the water volume. It is also interesting to observe that the solidification time is not that much influenced with the increase in water height, however it does increase significantly when diameter is increased. As in the case of 20×20 and 20×30 or 30×30 and 30×40 containers the change in solidification time is little, i.e. the change in solidification time in 20×20 and 20×30 is 15 s and the change in solidification time for 30×30 and 30×40 is 20 s. With the passage of time, the temperature gradient decreases. Also, the convection due to buoyancy helps in decreasing the temperature gradient which ultimately results in slow temperature change with time. But, once the temperature reaches the phase change temperature of water, there is a sudden change in temperature which is a typical characterstic of the phase change process.

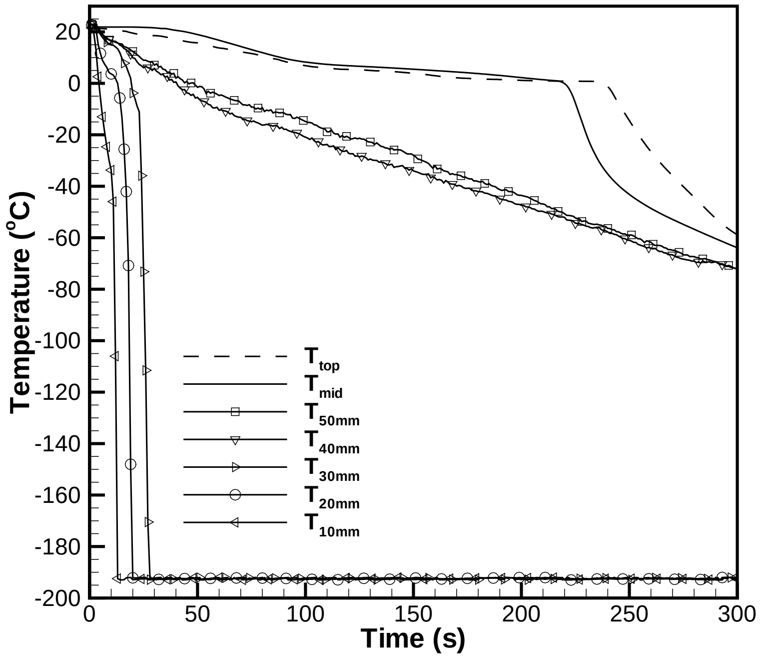
After the solidification of water, maintain the level of the liquid nitrogen for some time and found that the temperature profile of ice decrease suddenly for all the containers. This suddenly temperature drops happened because the conductivity of the ice increase with the decrease the temperature of ice.

4.1.2 Temperature variation of copper containers and PCM

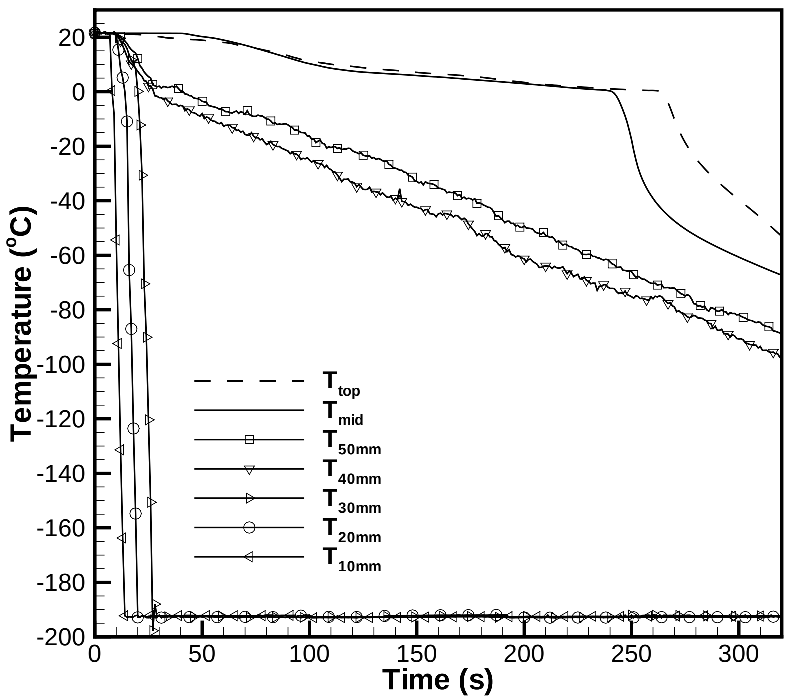
The figure 4.2 represents the temperature variation of all the thermocouples placed at 10, 20, 30, 40, and 50 mm from the base and axially at the mid and top of the PCM for 20×20 and 20×30 copper containers. It is seen that the copper container takes a few seconds (around 30 s) to reach the liquid nitrogen temperature, i.e. half of the copper container from the base is always in contact with the liquid nitrogen, but once the liquid nitrogen reaches the 30 mm from the base, then the level of liquid nitrogen is maintained throughout the experiment as represented by the graph of T_{10}, T_{20} and T_{30}. As the surface of copper container above 30 mm is always in contact with the vapour of liquid nitrogen, the temperature variation is approximately linear, which can be visualised from the graph of T_{40} and T_{50}.

4.1.3 Temperature variation of top and mid thermocouple

Figure 4.3 shows the temperature variation with time at the axial mid and top locations for the case of container size 30×30, 30×40 and 40×40. The two temperatures are denoted by T_{mid} and T_{top} respectively. As soon as the liquid nitrogen is poured into main container, whole of the copper block is engulfed by the nitrogen vapour. This induces a strong convective heat transfer through the top surface. As a result, the temperature at the top reduces much faster than the temperature of the axial mid location, which can be clearly seen in the figure 4.3. Generally, the total solidification time increases with the increase in water volume as shown in table 4.1. However, it is interesting to ob-

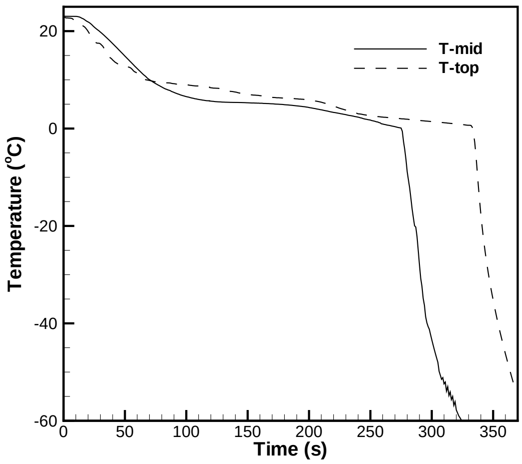


(a) Temperature history for 20×20 copper container

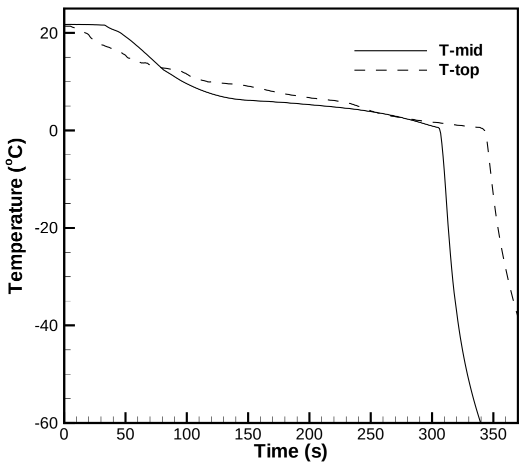


(b) Temperature history for 20×30 copper container

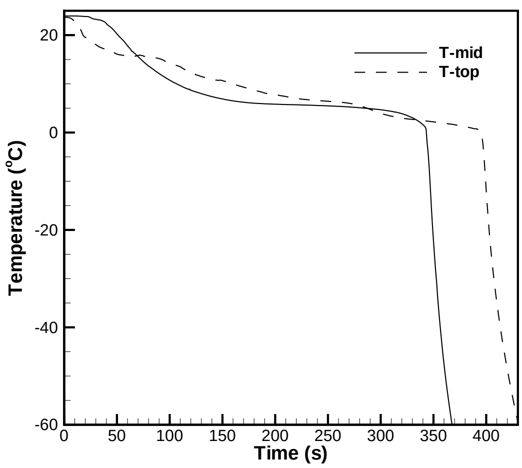
Figure 4.2: Temperature history for whole copper container and PCM



(a) Temperature variation of the PCM in a cavity of 30×30



(b) Temperature variation of PCM in a cavity of 30×40



(c) Temperature variation of PCM in a cavity of 40×40

Figure 4.3: Temperature variation of PCM at the Top and Mid of the axis in the cylindrical cavity of copper containers

Table 4.1: Total solidification time for all the container

containers	Time (s)
20×20	238
20×30	247
30×30	334
30×40	343
40×40	396

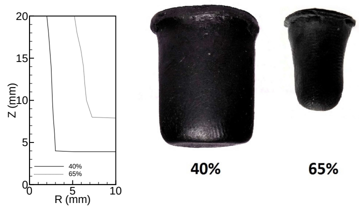
serve that the total solidification time changes marginally when the height of the cavity is increased. But, on the other hand, the total solidification time changes significantly when the diameter of the cavity is increased, keeping height fixed. For example, when cavity height is increased from 20 mm to 30 mm for the same cavity diameter of 20 mm, the solidification time increases by only 3.8% while it is only 2.7% when the cavity height is increased from 30 mm to 40 mm keeping cavity diameter fixed at 30 mm. But, this figure changes to 35% and 15% when the cavity diameter is changed from 20 mm to 30 mm for the same height of 30 mm and from 30 mm to 40 mm keeping height fixed at 40 mm.

4.2 Ice track in the copper containers

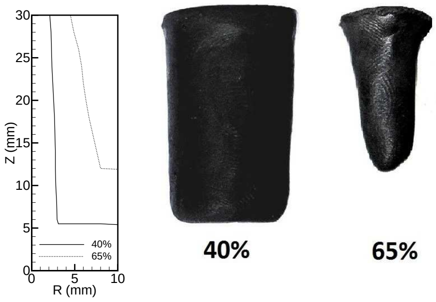
If the system is transparent like glass or acrylic sheet, then by using some light arrangement, it is easy to track the ice front. For example Lipnicki and Weigand [18] mixed Methylene blue to water so that the water-ice, interference observe clearly and additionally a neon light passed from the glass. The tracks of ice front are tough if the system is opaque, then going for some alternative for example [17] use syringe for removing liquid PCM from the mold at different time step during the experiment. This thesis also going for the alternative option and develop a new idea for trace the ice at different time step.

4.2.1 Ice track at 40% and 65% of solidification

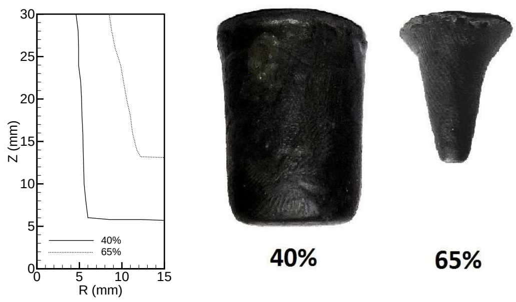
The ice tracking was done at 40 % and 65 % of the total solidification time. The graph was plotted for the thickness of ice in a two dimensional PCM domain through the use of the epoxy compound statue. Figure 4.4 presents the axisymmetric view of the ice



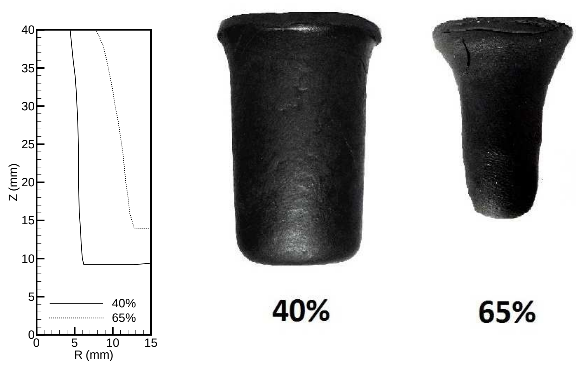
(a) Ice-track for 20×20 copper container at 40% and 65% solidification



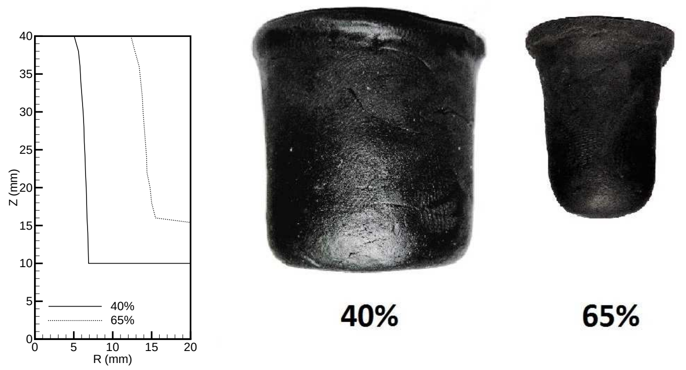
(b) Ice-track for 20×30 copper container at 40% and 65% solidification



(c) Ice-track for 30×30 copper container at 40% and 65% solidification



(d) Ice-track for 30×40 copper container at 40% and 65% solidification



(e) Ice-track for 40×40 copper container at 40% and 65% solid
cation

Figure 4.4: Ice thickness with epoxy compound statue at 40% and 65% of solidification

layer thickness at 40% and 65% of the total solidification time along with the epoxy compound statue which is also obtained after same duration. In the figure, the area under the solid and dotted lines represents, the thickness of ice at 40% and 65% of solidification of PCM respectively. According to 40% solidification result, the initial solidification process is mainly governed by the diffusion process. That is why the profile for the ice layer thickness is almost flat at the 40% of the solidification time. However, at this instant, the ice layer thickness at the base is more compared to the ice layer thickness across the height. The obvious reason is the higher conduction loss through the base compared to the loss through the circumference. The effect of convective heat transfer through the lateral surface to the nitrogen vapour is well reflected by a slightly slant vertical profile of ice layer at 40% of the solidification. But, the effect of convective heat transfer through the top surface is not that much significant. When the solidification process reaches towards 65% of the solidification time, the effect of convective heat transfer through the top is noticed for almost all the studied cases. This effect delays the solidification of water near the axial top location. It is also observed that the rate of solidification increases towards the end of the solidification process.

4.2.2 Variation of side ice thickness

The figure 4.5 and table 4.2 represents the variation of lateral thickness of ice at top

Table 4.2: Variation of side ice thickness in different cavity

Containers	40 % solidification		65 % solidification	
	Top ice thickness	Bottom ice thickness	Top ice thickness	Bottom ice thickness
20×20	2.1	2.8	5.2	7.25
20×30	2	3	4.5	8
30×30	4.6	6.2	8.5	12.2
30×40	4.4	6.4	7.5	12.8
40×40	5.05	7	12.34	15.5

and bottom locations in different containers at intermediate solidification. The horizontal ordinate represents the copper container, where 1, 2, 3, 4 and 5 represent the 20×20, 20×30, 30×30, 30×40 and 40×40 copper containers respectively. The solid line represents the condition at 40% of the solidification while dotted line represents the condition at 65% of the solidification. The minimum thickness is observed at the top

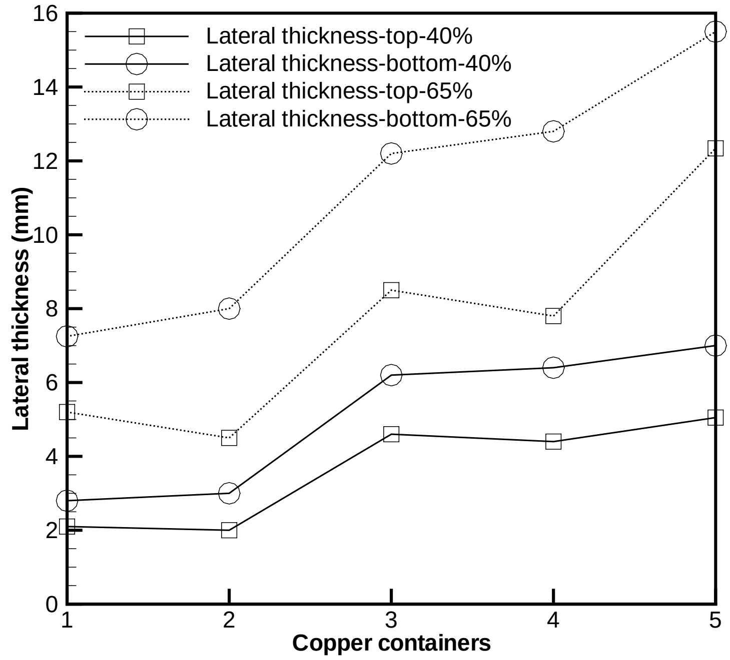


Figure 4.5: Variation of the lateral ice thickness while increasing the cavity dimension

from the side while the maximum is observed at the bottom. It is interesting to observe that the lateral top thickness reduces when the internal height of the copper container increases for the same internal diameter of the copper container while it increases if the internal diameter increases for the same height. But this effect is not noticed for the lateral thickness of ice at the bottom location. For this case, the thickness keeps on increasing with the increase in any one of the dimensions of the cavity.

4.2.3 Variation of base ice thickness

Similar trend of increasing ice layer thickness is also observed at the axial location. This trend is depicted on figure 4.6. In this figure also the horizontal ordinate represents the copper containers, i.e. 1, 2, 3, 4 and 5 represent the 20×20, 20×30, 30×30, 30×40 and 40×40 copper containers respectively.

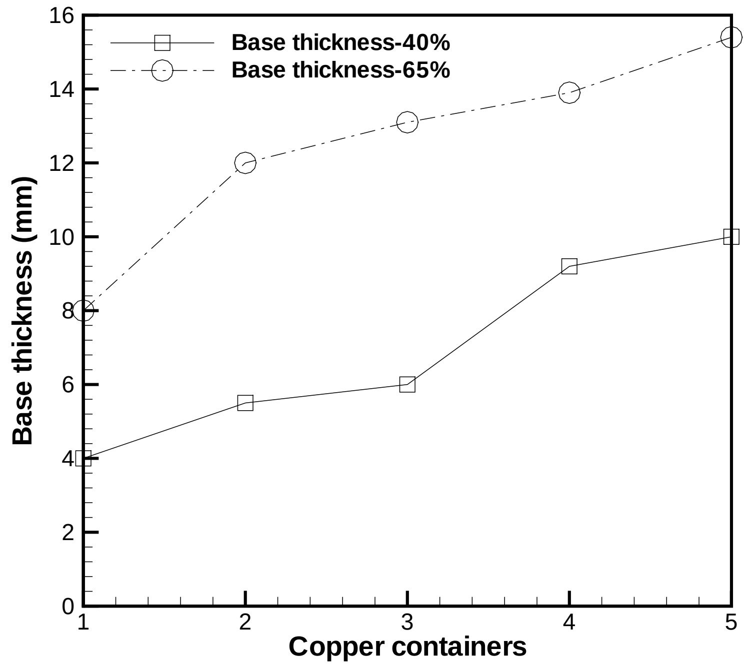


Figure 4.6: Variation of the base ice thickness while increasing the cavity dimension

4.3 Conclusion

It is concluded that

As the internal diameter is increased the solidification time is observed to be increased by significant value as compared to the increase in internal height.

It is interesting to note that as the internal height is increased, for constant internal diameter, lateral top ice thickness is reduced. But on the contrary as the internal diameter is increased, for constant internal height, the ice thickness is observed to be increased.

CHAPTER 5

Summary

This Experimental work mainly comprises of three sections —

(i) Design and development of experimental setup.

(ii) Studying the heat transfer and temperature variation in the PCM and periphery of copper blocks.

(iii) Developing an idea for measuring the ice thickness in the cavity at various time step during the solidification.

The main objective of this study is to measure the ice thickness experimentally. The ice thickness for the each container was recorded at the 40% and 65% of solidification and found that initially, uniform solidification takes place but after 65% of the solidification an inverted bell shape is observed. This proves that atmospheric convection dominates at the upper surface. The expansion of the ice looks like a hemispherical shape.

It is also found that when the depth increases from 20mm to 30mm for the same internal diameter (20mm), the solidification time increases by only 3.78% and when the depth increases from 30mm to 40mm, the solidification time increases by 2.69%. But when the internal diameter increases for the same height, there are more change in the solidification time. For example, as the internal diameter increases from $20mm$ to $30mm$ and $30mm$ to $40mm$ for the same height of $30mm$ and $40mm$ respectively, the change in the solidification time is 35% and 15% respectively.

5.1 Limitations and Recommendations

The experiment was done with the aim that the result can be used for cryo-preservation but experiment with the tissue was not done. Now it is very difficult to say that what happens when tissue is used in the PCM. The experiment is done in the open atmosphere, but when the tissue is to be used, it has to be in a closed environment. These results are valid under the specific boundary condition. The relation between depth and internal diameter of solidification is good when the copper container is in contact with extremely low temperature.

This experiment was done with the copper container, in future it can be done with some other material. The variation of the size of the container can be studied in detail. For ice tracking, some temperature sensing instrument can be used so that experiment can be done in one step and results are more accurate.

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